



New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis
CRM 116-A

Uranium (enriched) Metal Assay and Isotopic Standard

Certified Property Values

Amount Content	Value	Expanded ¹ Uncertainty	Isotope- Amount Ratio	Value	Expanded ¹ Uncertainty
g U•g ⁻¹ metal	0.99945	0.00014	$n(^{233}\text{U})/n(^{235}\text{U})$	0.0000003863	0.0000000086
			$n(^{234}\text{U})/n(^{235}\text{U})$	0.0115836	0.00000097
Molar Mass	Value	Expanded ¹ Uncertainty	$n(^{236}\text{U})/n(^{235}\text{U})$	0.0094713	0.00000077
g•mol ⁻¹	235.18572	0.00011	$n(^{238}\text{U})/n(^{235}\text{U})$	0.051277	0.000041
Isotope-Amount Fraction (•100)	Value	Expanded ¹ Uncertainty	Isotope Mass Fraction (•100)	Value	Expanded ¹ Uncertainty
$n(^{233}\text{U})/n(\text{U})$	0.00003603	0.00000080	$m(^{233}\text{U})/m(\text{U})$	0.00003570	0.00000079
$n(^{234}\text{U})/n(\text{U})$	1.08023	0.00089	$m(^{234}\text{U})/m(\text{U})$	1.07497	0.00088
$n(^{235}\text{U})/n(\text{U})$	93.2547	0.0038	$m(^{235}\text{U})/m(\text{U})$	93.1985	0.0038
$n(^{236}\text{U})/n(\text{U})$	0.88324	0.00071	$m(^{236}\text{U})/m(\text{U})$	0.88647	0.00071
$n(^{238}\text{U})/n(\text{U})$	4.7818	0.0036	$m(^{238}\text{U})/m(\text{U})$	4.8401	0.0037

¹ Expanded uncertainties for certified property values have a coverage factor of approximately 2.0 with the exception of the amount content value which has a coverage factor of 2.4 and the ²³³U values which have a coverage factor of 3.3 for isotope amount ratio, isotope-amount fraction, and isotope mass fraction.

Notes:

Certified Reference Material 116-A (CRM 116-A) is a uranium amount content and isotope-amount ratio standard intended for use in calibration of and/or quality control for uranium analysis methods. Each unit of CRM 116-A consists of a metal piece with a mass of approximately 1.1 grams. This CRM is not characterized for total quantity of material which may be somewhat greater or less than the nominal mass (between 1.0 g and 1.2 g).

CRM 116-A is a radioactive material and should be handled and stored under proper radiologically-controlled conditions at all times.

CRM 116-A units do not have an expiration date. To maintain the integrity of an unused unit, it should remain in the original packaging and should be stored in a dry, temperature controlled location.

Measurements for uranium amount content and isotope-amount ratios were performed on metal samples with a mass of 1.1 gram or greater. The homogeneity of uranium amount content or isotopic composition has not been assessed for metal pieces smaller than 1.1 gram. Prior to use, surface oxide must be removed to ensure accurate uranium amount content values. A suggested procedure is provided below.

Suggested Preparation Procedure for Achieving Accurate Mass and Amount Content Values

1. Cover the uranium metal sample in 8 mol•L⁻¹ nitric acid for 10-20 minutes to remove all visible surface oxides.
2. To minimize oxidation of the sample and ensure an accurate determination of uranium metal mass, the following steps should be performed immediately following Step 1.
 - 2.1 Thoroughly rinse the metal piece with distilled, deionized water.
 - 2.2 Remove excess water by thoroughly rinsing the metal piece with pure acetone.
 - 2.3 Allow the acetone to evaporate (30 – 60 seconds is typically sufficient).
 - 2.4 Perform a weighing of sufficient accuracy and precision for user's need.

Description:

The CRM 116-A metal pieces are machined metal cylinders. The stock material for the CRM was obtained from a single casting of a HEU right-annular cylinder of metal. Several wedges of material were cut from the annular cylinder and machined into rods which were stamped into narrow-diameter rods. The rods were then machined to shape and cut into the individual 1.1-gram metal cylinders that comprise each CRM 116-A unit.

Uranium amount content for CRM 116-A was determined by the NBL High Precision Titrimetric method using CRM 99 Potassium Dichromate Oxidimetric Standard as the titrant. The CRM 112-A Uranium Metal Assay and Isotopic Standard was used as a control to verify performance of the measurement system. Traceability of the measurements is primarily established by direct determination of uranium amount content based on the titration of uranium using CRM 99 Potassium Dichromate Oxidimetric Standard. CRM 99 was calibrated against CRM 112-A which, in turn, was originally provided by the National Bureau of Standards (now known as the National Institute of Standards and Technology) as SRM 960.

A detailed thermal ionization mass spectrometry measurement campaign was performed on CRM 116-A to determine uranium isotope-amount ratios and uncertainties. Mass discrimination calibrations were performed on a sample turret basis using multiple measurements of NBL Uranium Isotopic Standards U900 and U930-D. Analyses of CRM U970 Uranium Isotopic Standard were performed to verify that mass spectrometric measurements were in control. Traceability of the isotope-amount ratio measurements for CRM 116-A was established by calibration of the mass spectrometers using combined measurements of CRMs U900 and U930-D Uranium Isotopic Standards. CRM 900 was originally provided by the National Bureau of Standards (now known as the National Institute of Standards and Technology) as SRM U900. U930-D is directly traceable to National Bureau of Standards SRM U930 Uranium Isotopic Standard.

Measurement Uncertainty:

Reported numerical uncertainties for values are expressed as expanded uncertainties ($U = k \cdot u_c$) at the 95% level of confidence, where the expanded uncertainty (U) is the product of the combined standard uncertainty (u_c) and a coverage factor (k). The last figure in reported values and uncertainties is provided for information purposes and is not intended to convey a significant degree of reliability. The isotope-amount and weight fraction values and uncertainties are provided primarily for information purposes. To assure proper uncertainty propagation, it is recommended that isotope-amount ratios and associated uncertainties be used for calculations incorporating CRM 116-A values.

Uncertainties were determined according to the protocols outlined in JCGM 100:2008 *Guide to the Expression of Uncertainty in Measurement*. The combined standard uncertainties for attribute values consist of Type A and Type B components. The Type A uncertainty components for amount content is derived from the standard deviation of high precision titrations performed on 1.1 g U metal samples and the standard uncertainty for the primary analytical amount content measurements, which utilized 3-g U metal samples. The Type B component is the combined standard uncertainty of the CRM 99 oxidimetric standard. The Type A components for isotope-amount ratios are derived from standard deviations associated with isotopic ratio measurements of the samples and the $n(^{238}\text{U})/n(^{235}\text{U})$ ratio of NBL CRMs U900 and U930-D. Type B components are based on the combined standard uncertainties for the $n(^{238}\text{U})/n(^{235}\text{U})$ ratios of CRMs U900 and U930-D and components to account for additional sources of uncertainty associated with background corrections and analytical biases. Isotope mass fractions incorporate an additional Type B component associated with the uncertainty of the atomic mass for the U isotopes. The coverage factor (k) for each expanded uncertainty is based on the effective degrees of freedom for that quantity and is the Student's t-factor necessary to provide a 95% level of confidence ($k \approx 2.0$ for the values cited in this certificate except for the amount content value with $k = 2.4$ and the ^{233}U isotope amount ratio, amount fraction, and mass fraction which have coverage factors of $k = 3.3$). A more detailed explanation of measurement uncertainty can be obtained upon request from NBL.

References:

Bureau International des Poids et Mesures (BIPM), Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement, JCGM 100: 2008.